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Wear Behaviour of Nickel Coatings Reinforced by Recycled Quarry Dust: Influence of Current Density

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ABSTRACT

Nickel coatings incorporated with quarry dust were synthesized through direct current electrodeposition from a nickel Watt's bath. The study explored the effects of varying current densities on the surface morphology and wear behaviour of the nickel-quarry dust (Ni-QD) composite coatings deposited on a high-speed steel (HSS) substrate. Quarry dust was chosen as a reinforcement material due to its high silica and alumina content, which enhance the properties of the coating. To achieve finer particle size, the quarry dust was subjected to ball milling before electrodeposition. The study tested a range of current densities from 2 to 8 A/dm², as different current densities produce different results. The composite coatings were characterized using a Scanning Electron Microscope (SEM) and their wear resistance was evaluated through pin-on-disk test. The results indicated that increasing the current density enhanced the wear resistance of the coatings. Coatings produced at high current densities displayed a colony-like structure, demonstrating the impact of deposition conditions on colony size relative to current density. Ni-QD composite coatings created at 6 and 8 A/dm² resulted in smoother and narrower wear scars with minimal scratching, attributed to the low surface roughness of the coatings.

1. Introduction

The quarry industry is crucial for Malaysia's development, supplying essential raw materials for construction, building, and manufacturing sectors, thereby significantly boosting economic growth. Malaysia benefits from diverse natural aggregate resources across Peninsular and East Malaysia. According to the Department of Statistics Malaysia (DOSM), this mining sub-sector has grown rapidly, with gross output rising from RM1,528.3 million in 2010 to RM3,547.7 million in 2015 [1]. However,

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a critical and contentious issue within Malaysia's quarry industry is its environmental impact, particularly concerning natural aggregate and limestone production. As highlighted by Sridharan *et al.,* [2] approximately 20-25% of each crusher unit's output ends up as quarry dust waste. Efforts are underway to address this environmental concern by repurposing these industrial wastes into usable raw materials for practical applications, thereby reducing their environmental footprint.

Recent developments in nickel-based composite electrodeposition have focused on incorporating ceramic particles like SiC, Cr_2O_3, TiO_2, Al_2O_3 and WC into the nickel matrix to enhance the mechanical strength, corrosion resistance, and tribological performance of the coatings. Key factors influencing these coatings' properties include the type, size, concentration, and uniform distribution of these embedded particles. However, the widespread adoption of such coatings is limited by the high cost of ceramic particles and the required process adjustments for their integration into the metal matrix. To address these cost challenges, there is growing interest in exploring more economical reinforcement materials such as quarry dust, which contains high levels of SiO_2, as alternatives for ceramic reinforcement particles in the development of metal matrix composites (MMCs). Numerous studies have been conducted to enhance the mechanical, tribological, and corrosion resistance properties of these composites [3-8].

It has been demonstrated that adding quarry dust particles to metal matrices significantly enhances the final composites' wear resistance and mechanical characteristics in comparison to the base alloy. For instance, adding 7.5% of quarry dust to the metal matrix can increase the composite's tensile strength by 15% compared to the base alloy [9]. This improvement is a clear indication of the reinforcing effect of quarry dust particles within the metal matrix. Further evidence of the beneficial impact of quarry dust is provided by Ramesh *et al.,* [9], who demonstrated that a 10% quarry dust reinforcement in aluminum-quarry dust composites results in a maximum hardness of 82 BHN. This represents a 13.7% increase in hardness compared to the base alloy matrix. This significant enhancement in hardness is attributed to the uniform dispersion of the hard quarry dust particles, which act as obstacles to dislocation movement, thereby strengthening the composite. Moreover, increasing the quarry dust content from 10 to 20 wt% has been found to improve the composite's wear resistance significantly [10]. This improvement is due to the increased presence of hard, wear-resistant particles within the matrix, which reduces the material loss during wear. The enhanced wear resistance makes these composites particularly suitable for applications requiring high durability and longevity.

The usage of quarry dust as a reinforcement in MMCs for coating application has been expanded as a result of the considerable benefits it has demonstrated in improving the mechanical characteristics and wear resistance of MMCs. This expansion of its application underscores its capability to strengthen coatings, making them more resilient and durable against mechanical stresses and wear.

One of the simplest and most versatile methods for fabricating metal matrix composites (MMCs), particularly those with particles added to the matrix, is the electrodeposition process. This technique involves the deposition of a metal matrix onto a substrate while simultaneously incorporating reinforcing particles. The characteristics and quality of the electrodeposited MMCs are directly influenced by various parameters of the electrodeposition process. Key parameters include the composition of the electrolyte, the pH level of the electrolyte, the current density used during deposition, the duration of the deposition process, the type of reinforcement particles used, and their concentration within the electrolyte. Among these factors, the current density of the electrodeposition process plays a crucial role [11,12]. It significantly affects the chemical composition, microstructure, as well as the mechanical and corrosion properties of the resulting MMCs. Adjusting the current density can lead to notable variations in these properties, thus making it a critical parameter for optimizing the performance and characteristics of the electrodeposited MMCs.

Limited research has explored the effects of quarry dust reinforcement on composite coating properties. This study focuses on nickel-quarry dust (Ni-QD) composite coatings fabricated via electrodeposition from nickel Watt’s electrolyte using different current density. The research comprehensively examines the impact of these current density on the surface morphology and wear characteristics of Ni-QD composite coatings. Findings indicate that coatings deposited at higher current density exhibit enhanced wear resistance, suggesting potential applications for effective wear protection.

2. Methodology

The high-speed steel substrate, measuring 40 x 30 x 3 mm, was initially ground using silicon carbide papers of varying grit sizes—specifically, 240, 400, 600, 800, and 1200. Subsequently, the substrates underwent a cleaning procedure involving rinses with ethanol and distilled water. Following preparation, the substrate was subjected to electrodeposition with Ni-QD composite coatings under different current densities. The essential chemical composition and operational specifications required for the electrodepositing of the Ni-QD composite coating onto the substrate are listed in Table 1. Quarry dust was collected from a quarry in Negeri Sembilan and ground using planetary ball mill equipment with a ball-to-powder ratio of 10:1 for three hours at 350 rpm. A schematic diagram of the composite coating's electrodeposition process is shown in Figure 1.

Particle size analysis, X-ray fluorescence (XRF), and scanning electron microscopy (SEM) were used in a comprehensive investigation to characterise the quarry dust particles. SEM was used to examine the surface morphology of the Ni-FA composite coatings. Using a pin-on-disk test, the wear performance of the composite coatings was assessed. During this test, a 10 mm diameter stainless steel ball with a hardness of 60 HRC was pressed against the samples under a constant 10 N load at a frequency of 5 Hz. A 2.69 mm stroke length and a 1500 second sliding time were used. Every testing cycle employed a fresh stainless-steel ball, and the tests were carried out at room temperature. In order to fully evaluate the wear behaviour of the composite coatings, the resulting wear scars were subjected to SEM, energy-dispersive X-ray spectroscopy (EDS), and surface profilometry analysis.

Table 1
Chemical formulation and operational parameters of the modified nickel Watt’s bath solution

Bath composition	Concentration (g/l)
Nickel sulphate hexahydrate	200
Nickel chloride	20
Boric acid	30
Sodium citrate	30
Electrodeposition operating conditions	
Temperature	40°C
Deposition time	60 min
Current density	2, 4, 6, 8 A/dm^2
Concentration of quarry dust	45 g/l

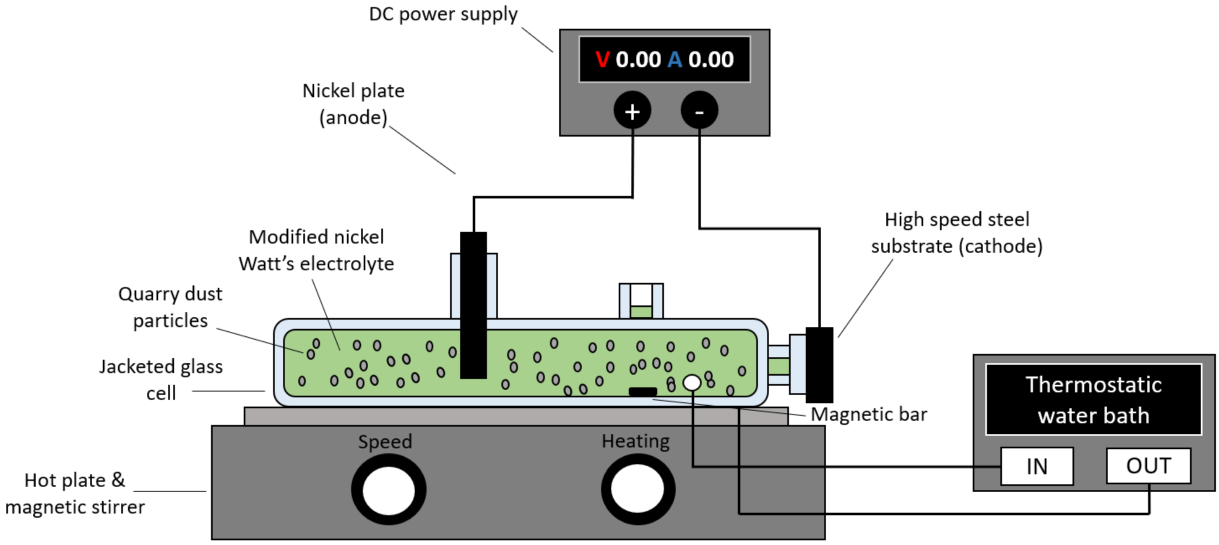


Fig. 1. Illustrative diagram of the electrodeposition setup

3. Results

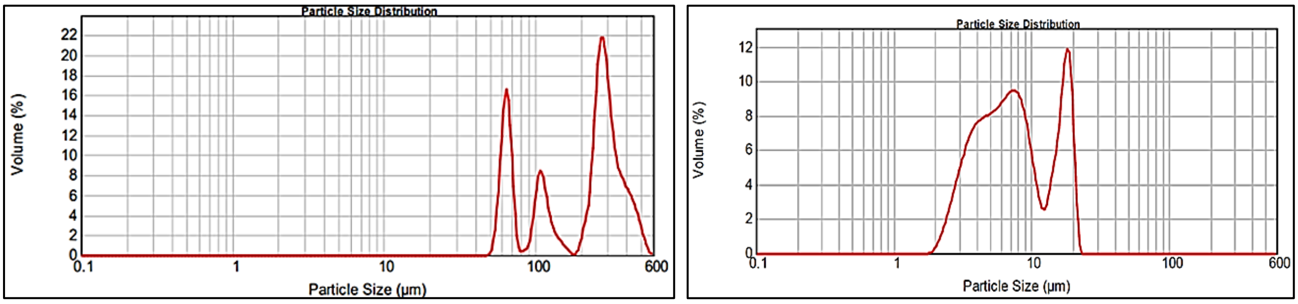
3.1 Characterization of Quarry Dust Particles

The elemental composition of quarry dust particles that underwent XRF analysis is displayed in Table 2. Phosphorus pentoxide (P_2O_5), magnesium oxide (MgO), sodium oxide (Na_2O), potassium oxide (K_2O), alumina (Al_2O_3), calcium oxide (CaO), ferric oxide (Fe_2O_3), sulphur trioxide (SO_3), titanium dioxide (TiO_2), and magnesium oxide (MgO) were all found in the analysis. The quarry dust particles had a high concentration of SiO_2 and Al_2O_3, two hard oxides that are frequently used as reinforcements in composites, according to the XRF results. This result is in line with research conducted by Cohen *et al.,* [13] and Khalid *et al.,* [14], who used quarry dust to create a sustainable alternative building material.

Table 2
The elemental composition of quarry dust particles as determined by XRF method

Element	Concentration (wt%)
SiO_2	72.6
Al_2O_3	15.1
K_2O	4.9
Na_2O	3.0
Fe_2O_3	1.9
CaO	1.1
MgO	0.8
TiO_2	0.3
SO_3	0.2
P_2O_5	0.1

Figure 2 presents the particle size distribution of the quarry dust in its initial state and after undergoing a 3-hour ball milling process at 350 rpm. Initially, the quarry dust showed primary grain sizes ranging approximately from 0.810 µm to 362.148 µm (Figure 2(a)). Following the ball milling process at 350 rpm, the particle sizes of the quarry dust were reduced, ranging from 1.684 µm to 15.157 µm compared to the original size distribution (Figure 2(b)). This reduction in particle size is attributed to the collision between the balls and the quarry dust particles during the milling process, resulting in finer particles.



(a) (b)

Fig. 2. Quarry dust particle size distribution (a) As received (b) Following ball milling

The results regarding the particle size distribution align with the SEM morphology of both samples, depicted in Figure 3. The SEM images clearly reveal that the quarry dust particles exhibit irregular and angular shapes with diverse sizes. Following the ball milling process, there is a noticeable refinement in the quarry dust, indicating a reduction in particle size. The results regarding the particle size distribution align with the SEM morphology of both samples, depicted in Figure 3. The SEM images clearly reveal that the quarry dust particles exhibit irregular and angular shapes with diverse sizes. Following the ball milling process, there is a noticeable refinement in the quarry dust, indicating a reduction in particle size.

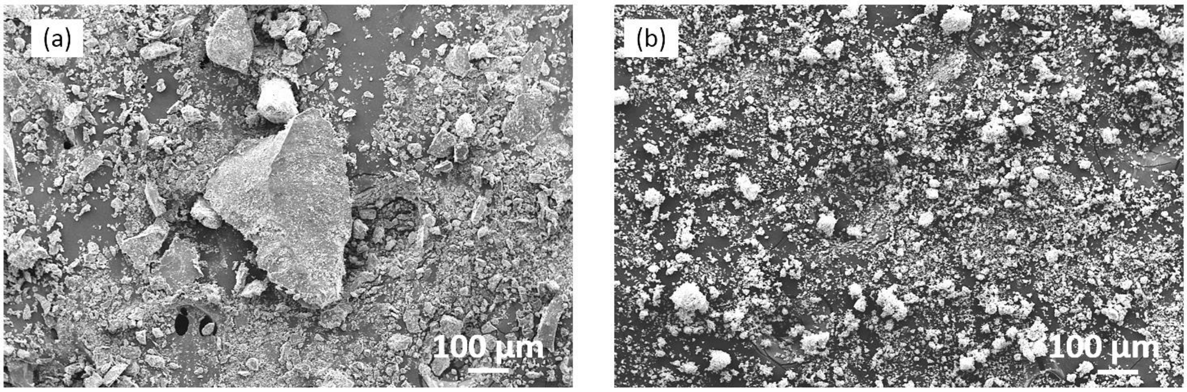


Fig. 3. SEM images of different conditions for quarry dust (a) As received (b) Following a three-hour ball milling process

3.2 Surface Morphology of Ni-Quarry Dust (Ni-QD) Composite Coatings

Figure 4 illustrates the surface characteristics of the composite coatings deposited at current densities of 2, 4, 6, and 8 A/dm^2. The coatings exhibit a transition in morphology from a compact and smooth surface at lower current densities to a less compact and coarser texture at higher current densities. This observation aligns with findings by Guo *et al.,* [15], who noted variations in deposition rates across different regions due to differing conductivities between alumina particles and the metallic matrix, leading to an uneven electric field distribution on the surface. At higher current densities (6 and 8 A/dm^2), these differences in deposition rates become more pronounced, resulting in a coarser surface morphology (Figures 4(e) and 4(g)). Additionally, it has been reported that increased hydrogen bubble formation on the coating surface at higher current densities reduces compaction and increases surface roughness [16].

In addition, Figures 4(e) and 4(g) clearly demonstrate that at high current densities, the surface morphology of the coating exhibits a colony-like structure, illustrating the impact of deposition conditions on colony size as a function of current density. At a lower current density (6 A/dm^2), colonies were approximately 7 µm in size, whereas at higher current density (8 A/dm^2), colony size gradually increased to 50 µm. This trend is also evident in the 3D surface profiles shown in Figure 5, illustrating a distinct colonial surface profile at a current density of 8 A/dm^2 (Figure 5(d)).

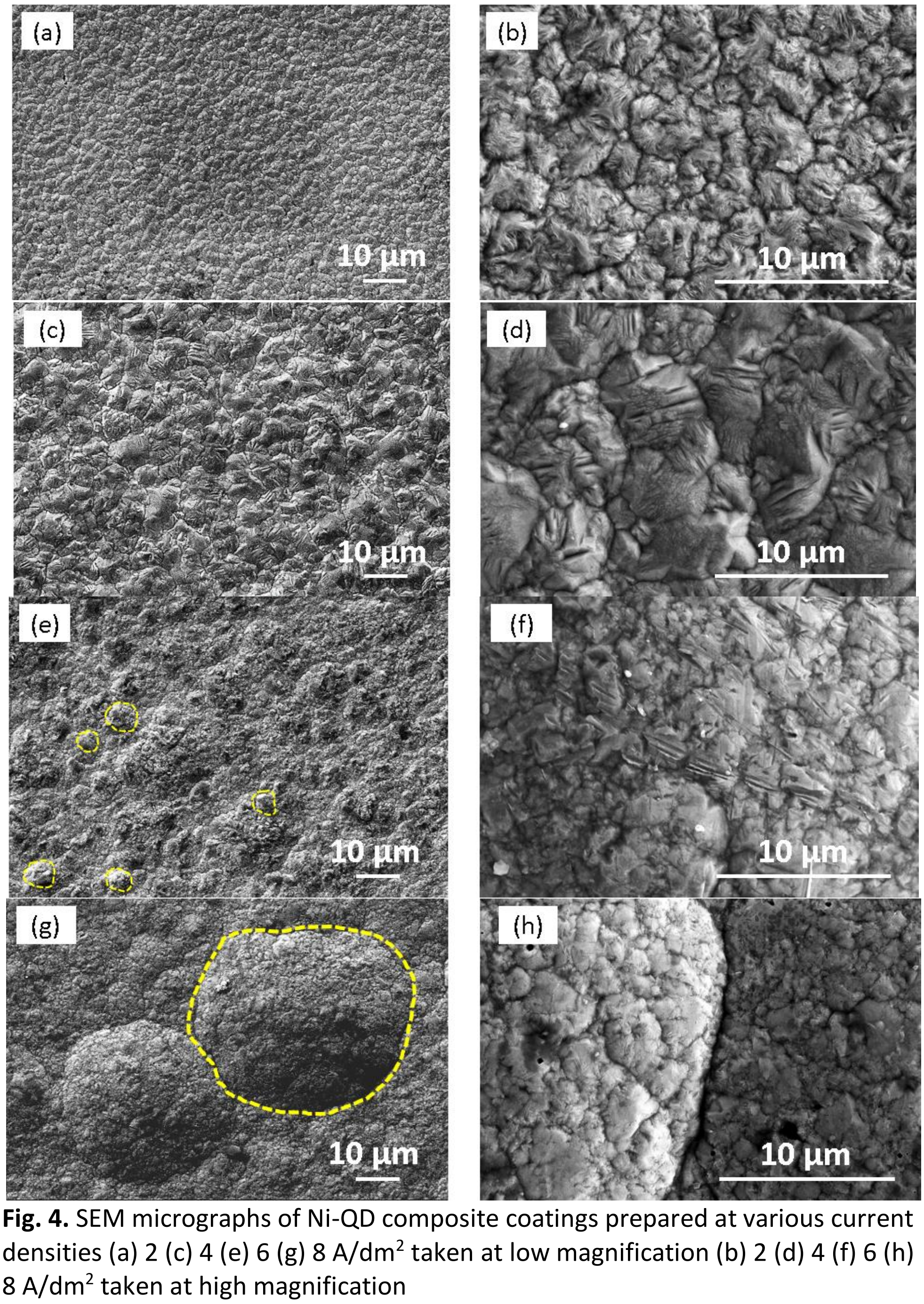


Fig. 4. SEM micrographs of Ni-QD composite coatings prepared at various current densities (a) 2 (c) 4 (e) 6 (g) 8 A/dm^2 taken at low magnification (b) 2 (d) 4 (f) 6 (h) 8 A/dm^2 taken at high magnification

In this study, the surface roughness (Ra) of electrodeposited Ni-QD composite coatings was assessed using a 3D non-contact profilometer. The results of these roughness measurements are presented in Figure 5, which demonstrates a clear relationship between the surface roughness and the microstructure of the surface, as shown in Figure 4, and its topography, as depicted in Figure 5.

At lower current densities, the surface roughness (Ra) increased because the colonies tended to grow more vertically than laterally, as illustrated in Figures 5(a) and 5(b). On the other hand, at higher current densities, the surface roughness (Ra) decreased, suggesting that there was more lateral growth compared to vertical growth of the colonies, resulting in larger colony sizes, as shown in Figures 5(c) and (d).

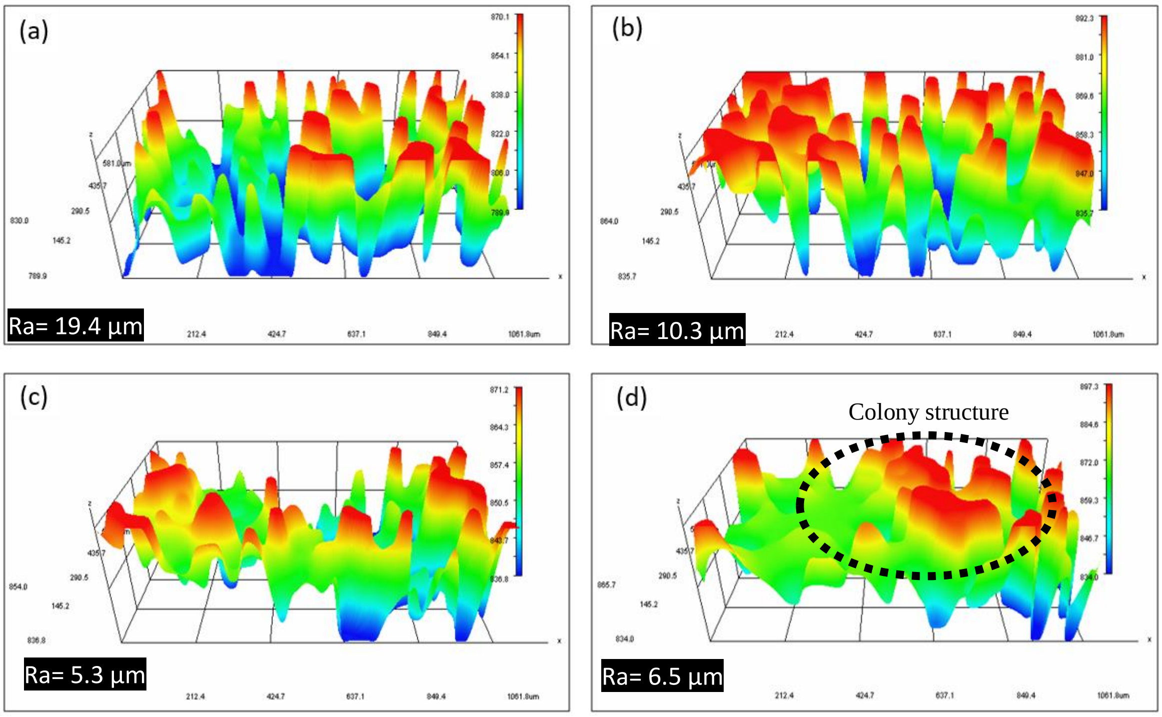


Fig. 5. 3D surface profiles of of Ni-QD composite coatings prepared at various current densities (a) 2 (b) 4 (c) 6 (d) 8 A/dm^2

3.3 Morphology Observation on the Wear Track of Ni- Quarry Dust (Ni-QD) Composite Coatings

To investigate the wear performance of Ni-QD composite coatings produced under different current densities, SEM images and corresponding EDS-mapping of elemental distribution on the worn surfaces are presented in Figure 6 and Figure 7, respectively. The wear scars, depicted in Figure 6, exhibit distinct abrasive grooves predominantly oriented parallel to the sliding direction. At 2 A/dm^2, the Ni-QD composite coating shows a wide and deep groove with visible furrows and areas of significant peeling, indicative of poor wear resistance (Figure 6(a)).

The poor wear resistance at low current density is attributed to the limited incorporation of quarry dust into the nickel matrix. This occurs because the slow movement of metallic ions toward the cathode results in insufficient metallic ions to embed the alumina particles. Consequently, when these particles reach the cathode surface, they are not adequately embedded and may become detached from the cathode surface [17]. Additionally, the thin coating produced at low current density easily peels off during wear testing due to its low thickness. According to Sherwin *et al.,* [18] and Prince Kumar and Gupta [19], the coating thickness increases proportionally with current density as the cathode potential and polarization steadily increase, thereby thickening the coating at the cathode. However, at high current densities, the uniformity of coating thickness deteriorates due to the accelerated electrodeposition process [20].

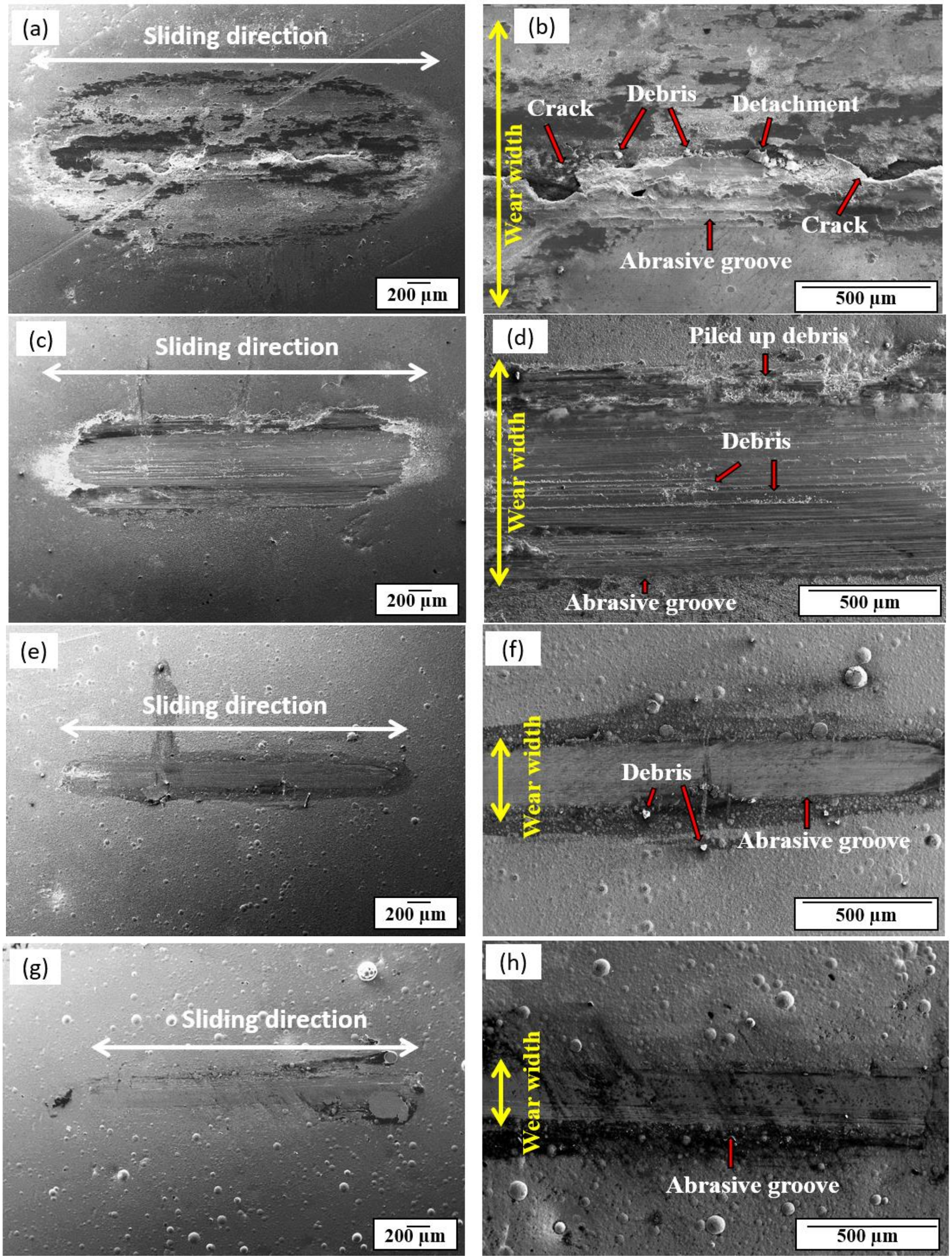


Fig. 6. SEM micrographs of wear tracks of Ni-QD composite coatings made at different current densities (a, b) 2 (c, d) 4 (e, f) 6 (g, h) 8 A/dm^2

In contrast, at higher current densities, notable differences in wear characteristics are observed compared to coatings produced at lower current densities. At 4 A/dm^2, the wear scar is narrower than that of the coating produced at 2 A/dm^2 (Figure 6(c)). Further increasing the current density to 6 and 8 A/dm^2 (Figures 6(e) and 6(g)) results in smoother and narrower wear scars with minimal scratching.

The measurements of the wear scar illustrated in Figure 6 were utilised to assess the level of wear damage because the uneven structure of the wear scar makes exact estimation of the worn volume difficult. Notably, for varying current densities, the wear scar dimensions on Ni-QD composite coatings vary dramatically. Poor wear resistance is indicated by the greatest and longest wear scar on the Ni-QD composite coating formed at 2 A/dm². On the other hand, when the substrate undergoes electrodeposition at higher current densities like 4, 6, and 8 A/dm^2, the wear scar's width and length considerably reduce. However, the wear scar length of coatings produced at 6 and 8 A/dm² does not significantly differ from one another. It has been proposed that wear resistance can be inferred from the depth and breadth of the worn region [21].

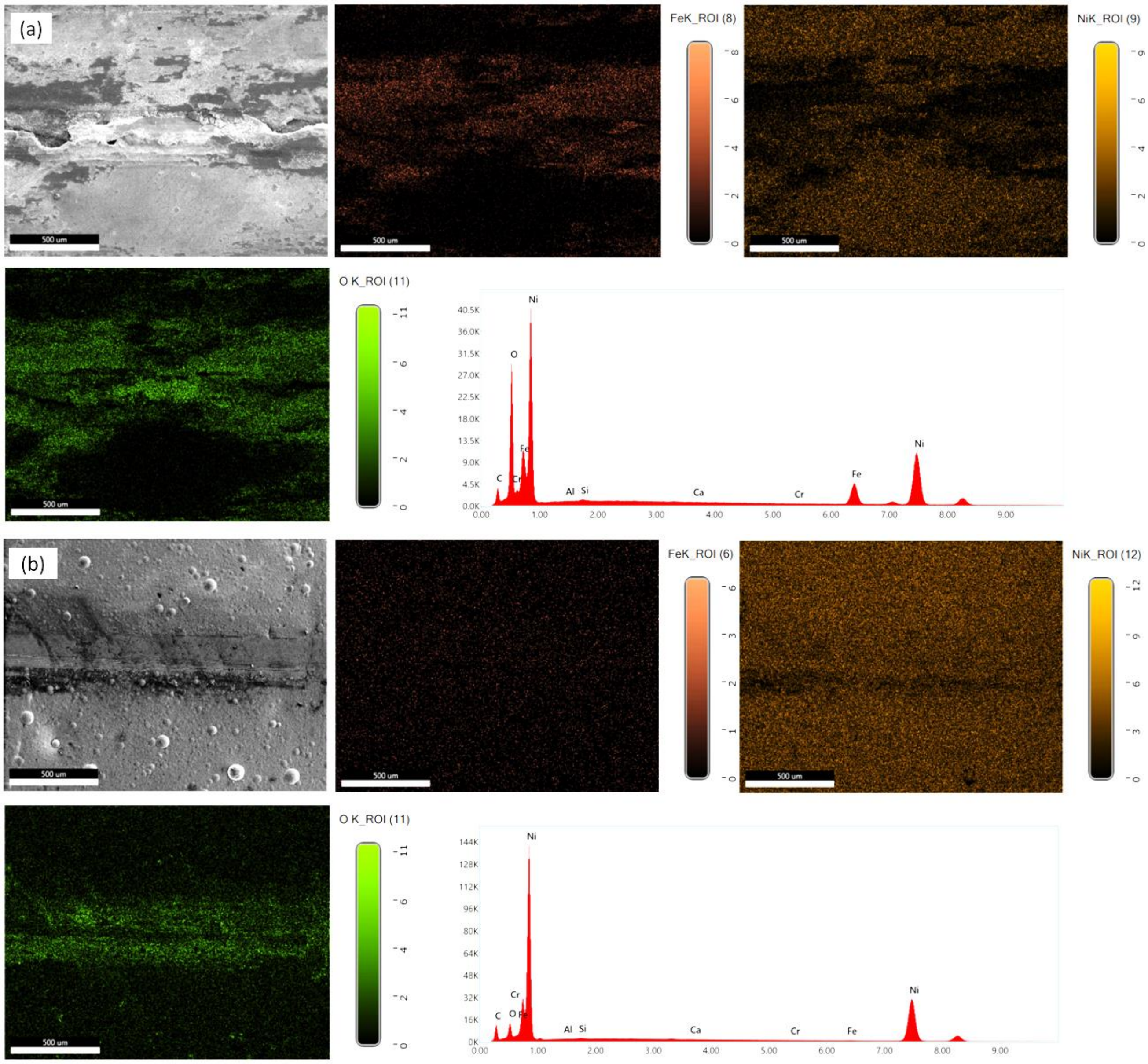


Fig. 7. Elemental mapping by EDS for Ni-QD composite coatings generated at different current density (a) 2 (b) 8 A/dm^2

As shown in Figures 6(a) and 6(b), the coating produced at low current density exhibits a wide and deep wear scar with distinct furrows, adhesive craters, and areas of significant peeling. This observation corresponds with the EDS mapping results of element distribution on the worn surface of the coatings, as illustrated in Figure 7. At low current density, the coating is easily ploughed and peeled, exposing detectable iron content along the wear track, which indicates the steel substrate is exposed as seen in Figure 7(a). Additionally, Table 3 indicates a substantial amount of oxygen on the wear tracks produced at low current density, suggesting that oxidation occurred during the wear of the composite coatings against steel due to frictional heating [12]. In contrast, the composite coating produced at high current density exhibits more uniform worn surface and less worn damage, indicating good wear resistance (Figure 7(b)).

Table 3
EDS elemental mapping of Ni-QD composite coatings generated at different current density

Element (mass %)	Current density (A/ dm^2)			
	2	4	6	8
C	13.2	15.0	19.7	21.5
O	17.2	14.2	2.3	3.8
Fe	10.6	0.6	0	0.2
Ni	58.4	69.5	77.7	73.8

4. Conclusions

Ni-QD composite coatings were successfully electrodeposited on a high-speed steel substrate at various current densities. The morphology and wear behavior of the Ni-QD composite coatings were influenced by current density. The main results are as follows:

i. Ni-QD composite coatings produced at high current densities of 6 and 8 A/dm^2 exhibit a colony-like structure, resulting in a coarser surface morphology.
ii. The surface roughness of Ni-QD composite coatings produced at low current density is higher than that at high current density due to the vertical growth of colonies at low current density, compared to lateral growth at high current density.
iii. The wear resistance of Ni-QD composite coatings electrodeposited at low current density is poor due to the low amount of quarry dust embedded in the nickel coating and the thinness of the coating.

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